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AMRL-TR-71-79

VACUUM DISTILLATION-VAPOR FILTERED CATALYTIC OXIDATION WATER RECLAMATION SYSTEM UTILIZING RADIOISOTOPES

R. J. HONEGGER

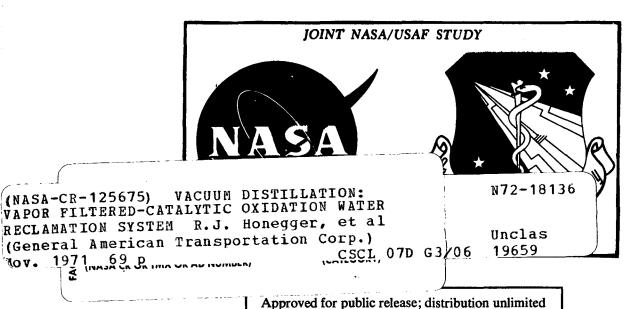
G. A. REMUS

E. K. KRUG

GENERAL AMERICAN RESEARCH DIVISION

GENERAL AMERICAN TRANSPORTATION CORPORATION

NOVEMBER 1971



AEROSPACE RESEARCH LABORATORIES
AIR FORCE SYSTEMS COMMAND
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AIR FORCE: 28-12-71/100

Security Classification			
DOCUMENT CONT	ROL DATA - R 8	& D	C 10 11 11 11 11 11 11 11 11 11 11 11 11
(Security classification of title, body of abstract and indexing 1. ORIGINATING ACTIVITY (Corporate author)	annotation must be e		
General American Transportation Corporation	n	UNCLASS	CURITY CLASSIFICATION
General American Research Division		2b. GROUP	
Niles, Illinois 60648		N/A	
3. REPORT TITLE VACUUM DISTILIATION - VAPOR FILTERED - C		DATION	
WATER RECLAMATION SYSTEM UTILIZING RADI	OLSOTOPES		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Final Report, March 1969 - June 1971			
5. AUTHOR(S) (First name, middle initial, last name) D. T. Wonggron			
R. J. Honegger G. A. Remus			
E. K. Krug			
6. REPORT DATE	78. TOTAL NO. OF	PAGES	7b. NO. OF REFS
November 1971	65		0
88. CONTRACT OR GRANT NO. F33615-69-C-1486 and NASA PR T-80498 b. PROJECT NO. 6373	GARD Repo	rt 1490-09	
c. Task 637305	9b. OTHER REPOR	T NO(S) (Any of	her numbers that may be assigned
d.	AMRL-TR	-71- 79	
10. DISTRIBUTION STATEMENT			
Approved for public release; distribution	unlimited.		
Supported in part by the National Aero- nautics and Space Administration (NASA) under NASA PR T-80498.	Aerospace M	edical Res edical Div	earch Laboratory ,, Air Force Systems rson AFB, Ohio 45433
The development of a functional mode system produces potable water by distilla perspiration condensate at the normal rat employed are vacuum distillation, vapor f and condensation. The system is designed distillation thermal input, and one 45-wa The additional electrical input power for 7 watthours per pound of processed water.	tion from the generated lateration, very to use four tt isotope for	e urine an by 4 men. apor phase 75-watt i or the cat	d respiration- Basic processes catalytic oxidation, sotope heaters for alytic oxidation unit.

The system is capable of collecting and storing urine, and provides for stabilizing the urine by chemical pretreatment. The functional model system is designed for operation in a weightless condition with liquid-vapor phase separators for the evapor ator still, and centrifugal separators for urine collection and vapor condensation. The system provides for storing and dispensing reclaimed potable water.

The system operates in a batch mode for 40 days, with urine residues accumulating in the evaporator. The evaporator still and residue are removed to storage and replaced with a fresh still for the next 40-day period.

The major system components, empty, weighed 75 lbs. The unoptimized system with framework, controls, piping and instrumentation weighed 148 lbs. The assembly occupied an envelope 29 by 24 by 60 inches.

Security Classification LINK A LINK B LINK C KEY WORDS ROLE wr ROLE ROLE Life Support Water Recovery Vacuum Distillation Vapor Filtration Catalytic Oxidation Zero Gravity

FOREWORD

This study was initiated by the Aerospace Medical Research Laboratory, Wright-Patterson Air Force Base, Ohio, and sponsored by the National Aeronautics and Space Administration. The work was performed in support of Project 6373, "Equipment for Life Support in Aerospace." The research was conducted by the General American Research Division of General American Transportation Corporation under Contract F33615-69-C-1486.

Mr. R. J. Honegger was the principal investigator for the General American Research Division. Mr. C. A. Metzger was the technical monitor for the Aerospace Medical Research Laboratory. The research was performed by the Chemical and Environmental Systems Group of the General American Research Division, under the direction of Mr. G. A. Remus. The work reported herein was initiated in March 1969 and was completed in June 1971.

This report is cataloged by the General American Research Division as Report 1490-0900.

This technical report has been reviewed and is approved.

CLINTON L. HOLT, Colonel, USAF, MC Commander Aerospace Medical Research Laboratory

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SECTION I

INTRODUCTION

The need for reclamation of water from urine on long duration multi-man spacecraft missions has been well established. Systems which carry out this recovery process must be reliable, have a minimum of moving parts, and have low power, weight and volume requirements. Many of the systems previously investigated for water reclamation utilize distillation for the basic water recovery process. For continuous and long duration operation, the management of accumulated residue is also an important consideration in the overall concept of the water reclamation processes.

Company sponsored investigative programs at the General American Research Division (GARD) in the past several years indicated the desirability of a static process for distillation of urine and management of accumulated residue. Advantages of the static process are low power requirement and high reliability accruing from the absence of major moving parts in the distillation system.

During the same period of time, tests were being conducted at the Aerospace Medical Research Laboratory to evaluate a system operating on the basic principle of static distillation. The Aerospace Medical Research Laboratory conceived, fabricated, and tested a static vacuum distillation system which employed a vapor filter and a catalytic oxidation unit to provide purified water vapor which could be condensed to provide potable water.

This basic design was used by GARD and modified as required to develop the system fabricated and tested in the program described in this report.

PROGRAM OBJECTIVES

The overall objectives of the program were to design, fabricate and evaluate an operating hardware model of a water reclamation system for processing the urine and respiration-perspiration condensate from four men during a 40-day period. The functional model was to demonstrate vacuum distillation, vapor filtration, vapor phase catalytic oxidation processes and vapor condensation necessary to reclaim potable water from urine. The system was to be designed for weightless operation, and system performance was to be demonstrated in a 30-day test.

The system was to process the daily waste products of four men composed of 3.50 pounds urine and 5.0 pounds condensate per man-day, and produce 7.50 pounds per man-day of potable water.

The recovered water was to meet U.S. Public Health Standards (1962) and/or the requirements established by the National Academy of Science, Space Science Board, Ad Hoc Panel on Water Quality Standards for Long-Duration Manned Space Missions," dated September 1967.

The complete system was to be designed for operation in a weightless condition, but was also to be compatible with testing at normal gravity.

SYSTEM PERFORMANCE

The reclamation system, consisting of a urine collection module, the main distillation and condensing subsystem, and product water storage module, was operated successfully in a 30-day performance test to provide water meeting the essential potability standards required. The system processed an average of 34 lb/day of combined urine and simulated respiration-perspiration condensate, to generate over 32 lb/day of product water. Most of the remaining feed collected in the evaporator as concentrated

residue, while some was also vented as vapor during elimination of noncondensibles from the condenser unit. The system has the capability to
operate for 40 days without replacement or emptying of the main evaporator
assembly.

A catalytic oxidation unit operating at 250F essentially eliminated trace organics in the distillation vapors. A small stream of air was admitted to the inlet of the oxidation unit to provide oxygen for the catalytic process.

Four electric heaters were used to distill the urine mixture; the heaters were housed in receptacles which will accept isotope heaters of the same size and shape as the electrical heaters.

The components of the system were designed for operation in a weightless condition, and insofar as is possible in normal gravity, were shown suitable for weightless operation. The urine mixture in the evaporator still was constrained by a wick assembly. A special hydrophobic membrane was utilized to separate distilled vapors from entrained liquid droplets during boiling.

Heat was transferred to the liquid in the wicks by a finned assembly connected directly to each heater housing. Evaporation occurred within the wick and solids eventually collected within the interstices of the wick. Capillary flow gradually slowed in the wick, and the level of urine liquid in the evaporator had to be raised above initial levels to restore the initial capillary flow rate.

Overheating occured in the evaporator when lowered capillary flow diminished the amount of liquid available for evaporation. Some decomposition of urine occured on overheating to temperature levels in the 120 to 140F range. The ammonia released raised the concentration of

ammonia in the product water to levels in excess of the generally desired range of 10 mg/l.

SYSTEM CHARACTERISTICS

The empty evaporator weighed approximately 45 lb. The remaining major components, namely the condenser, storage tanks, and collection and dispensing components, weighed approximately 30 lb. The complete assembled system with nonoptimized controls, instrumentations, and support frames weighed 148 lb.

During continuous operation the system power requirements, less the electrical heaters, averaged 7 watthours per pound of water recovered.

The system was assembled in framework designed for easy access to all components. The envelope for this configuration was 29" wide, 24" deep and 60" high. The estimated minimum envelope for an optimized package system is 20" wide by 20" deep by 30" high.

The assembled system is shown in Figure 1.

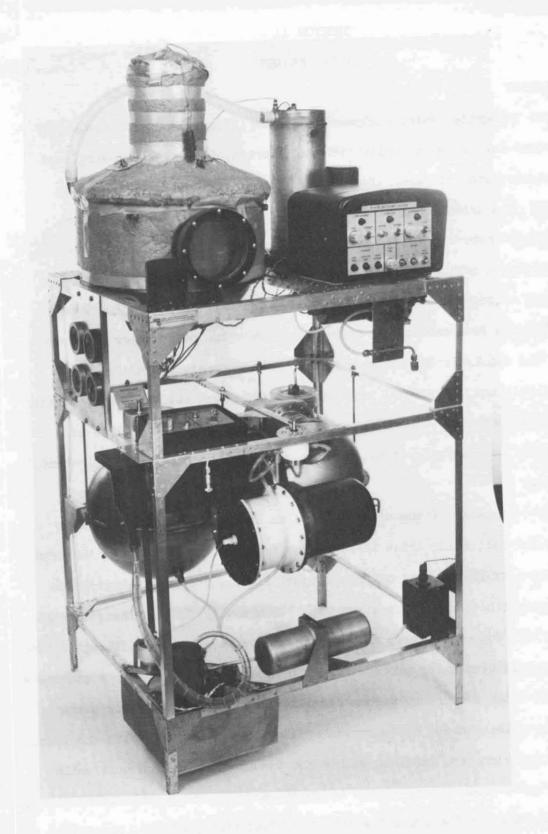


FIGURE 1 Water Reclamation System

SECTION II

SYSTEM DESIGN

DESCRIPTION

The Integrated Water Reclamation System is comprised of three major subsystems for (1) urine collection and storage, (2) water recovery, and (3) potable water storage. The urine collection and storage subsystem consists of a urinal, urinal flush tank, flush disinfectant reservoir, disinfectant metering valve, phase separator, acid pretreatment reservoir, pretreatment metering valve, blower, particulate filter, waste holding tank, and associated electrical controls. The water recovery subsystem consists of a feed control, evaporator assembly including vapor membrane filter and catalytic oxidation bed, condenser assembly including cooling coil and centrifugal separator, liquid level switch, and associated electrical controls. The potable water storage subsystem consists of a water pump, potable water tank, dispensing valve, and associated electrical controls.

The components of the urine collection and waste storage subsystem provide for collecting urine in zero gravity, chemically treating the urine by adding a small quantity of pretreatment liquid, receiving respiration and perspiration (dehumidification water) condensate from an Environmental Control/Life Support system, and storing the urine and dehumidification water mixture until needed for processing. The water recovery subsystem processes the waste water mixture by vacuum distillation, filtration, vapor phase catalytic oxidation and condensation of the product water. Solid and liquid residual products are retained within the vacuum distillation unit while gaseous waste products are vented to space vacuum. The potable water storage subsystem receives the product water at condensation pressure, pumps the

water to storage pressure, and stores and dispenses this water at normal room temperature.

SCHEMATIC FLOW DIAGRAM

The schematic flow diagram for the integrated water recovery system is shown in Figure 2. Referring to this diagram, the flow path through the system is described as follows.

Urine is admitted by the user into the direct connection type urinal where it is entrained in cabin air (transport media) entering through side bleed holes. The urine-gas mixture is drawn into a rotary separator by a blower which imposes a pneumatic drag on the liquid. An acid pretreatment solution is injected into the stream at the separator inlet to fix ammonia and retard putrefaction. Cabin air, which entered this loop through the urinal side bleed holes, passes through a filter to remove airborne microorganisms before the air is discharged back to the cabin. The dynamic head produced by the rotary separator provides for transferring the liquid to the holding tank. The blower and rotary separator operate only during micturition.

Respiration and perspiration condensate is admitted to the urinal flush tank from an external dehumidifier system. A mild disinfectant is injected simultaneously into the flush tank along with humidity condensate. Waste water from the holding tank is fed into the evaporator by a feed control unit.

In the evaporator, liquid is heated by fixed heat sources which are electric heaters but designed to be interchangeable in terms of physical size and performance with radioisotope heaters. The energy from the heat

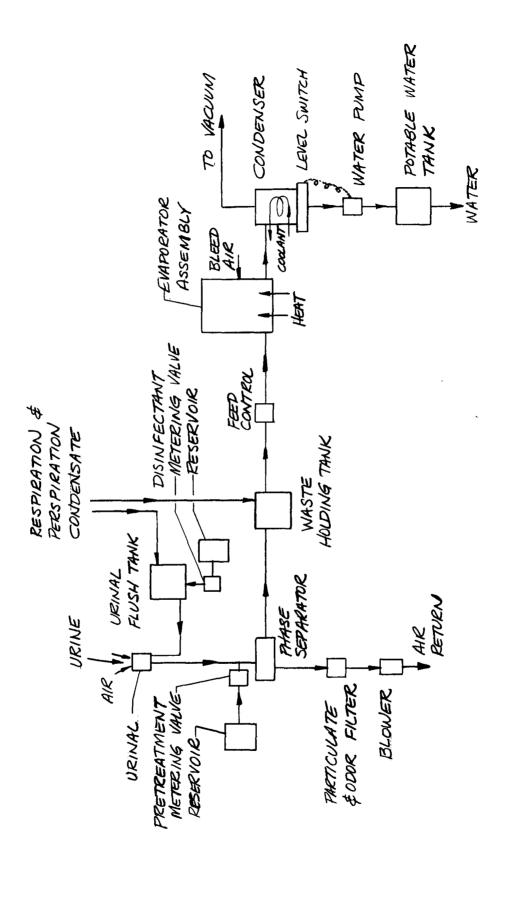


FIGURE 2 Schematic Flow Diagram

sources raises the temperature of the urine and water mixture to its vapor pressure and provides the heat of vaporization for distillation. Distilled vapors pass through a filter and through an entrainment separator before reaching the catalyst bed where entrained organic compounds are oxidized. Cabin gas is metered into the vapor process stream between the vapor filter and entrainment separator; the oxygen in this gas is required for the catalytic oxidation process.

The catalyst bed operates at 250 - 300F and is heated by an electric heater also interchangeable with a radioisotope. Vapor exiting from the catalyst bed passes through a particulate filter where catalyst fines, 200 mesh size and larger, are retained. The vapor then gives up a portion of its heat content through a recuperator in the evaporator before entering the condenser.

The condenser converts water vapor to liquid; the resulting potable water is transferred to the storage tank by a water pump. The condenser is cooled by an external coolant which absorbs the heat of condensation.

Noncondensable gases in the process stream pass from the condenser through a check valve to space vacuum. The system utilizes the pressure differential between the evaporator and condenser to act as the prime mover of the process stream.

The urine feed control operates intermittently to feed urine at a constant rate to the evaporator and is actuated by a timer. The condensate water removal pump also operated intermittently but is actuated by a liquid level control measuring the level of water in the condensate centrifugal separator.

The evaporator still is sized to operate for 40 days; during this period the liquid and solid residue from the waste mixture accumulate and

and remain in the still. After 40 days operation, the still and membrane assembly are removed completely from the system and another evaporator still and membrane are installed for the next 40 days of operation.

SYSTEM COMPONENT DESCRIPTION

Urinal (Figure 3)

The urinal is fabricated from machined aluminum which is hard-coat anodized and Teflon-coated. It incorporates a silicone rubber cuff seal which is dilated during the installation of the seal onto the urinal funnel. Four color coded seal assemblies provide individual cuff seals for each member of a four-man crew. The urinal is connected to a phase separator by a flexible hose. The hose is wire reinforced with a silicone wrap outer cover over a smooth bore Teflon liner.

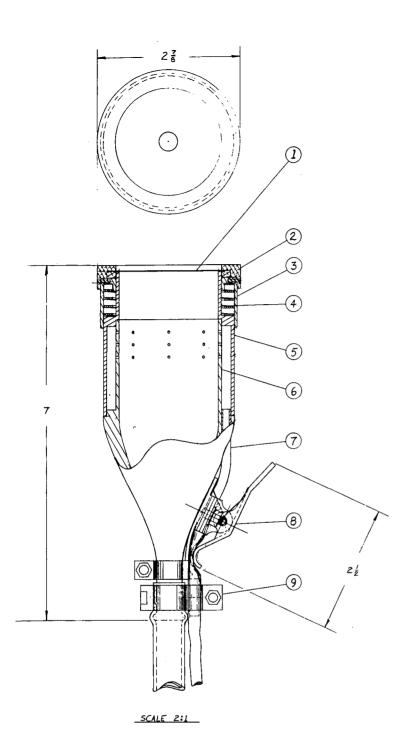
A spring loaded pinch valve is mounted at the base of the urinal. The valve may be depressed with the thumb or index finger to provide flush water for the user. Releasing the pinch valve lever stops the flush water.

Urinal Flush Tank (Figure 4)

The urinal flush tank is a cylinder containing a spring loaded diaphragm for zero gravity operation. The tank has a capacity of 900 cc. The diaphragm material is Viton A and metal parts are anodized aluminum.

Flush Disinfectant Reservoir (Figure 4)

This component is identical in construction to the flush tank described above.



			TITLE OR DESCRIPTION	70
Ī	`	1490-3100	DIAPHRAGM , SILICONE	Ī
1	Γ	1490-3200	DYAPHRAGM HOLDER ASSY	2
1		1490-3300	URINAL SLIDE ASSY	3
1		リナンひじつせいひ	SPRING, SLIDE ASSY RETURN	4
1	Γ		OUTER SHELL, URINAL	5
1		1490-3600	BODY , URINAL	6
1		1 19 0-3700	SUPPY TUBE URINAL DISINFECTANT	7
1			VALVE ASSY., URINAL DISINFECTANT LINE	ε
1			DUAL HOSE CLAMP ASSY	9

FIGURE 3 Urinal

FIGURE 4 Urinal Flush Tank

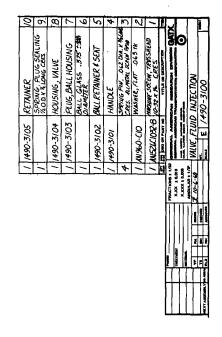
Disinfectant Metering Valve (Figure 5)

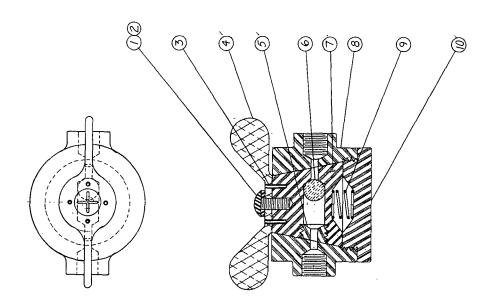
This valve consists of a tapered plug valve, with a ball moving back and forth within the plug. The ball travels a predetermined distance when the handle is rotated 180° and dispenses the volume of fluid equivalent to the ball travel and the area of its diameter. The injector body and plug are fabricated of Teflon; the ball is Hastelloy C.

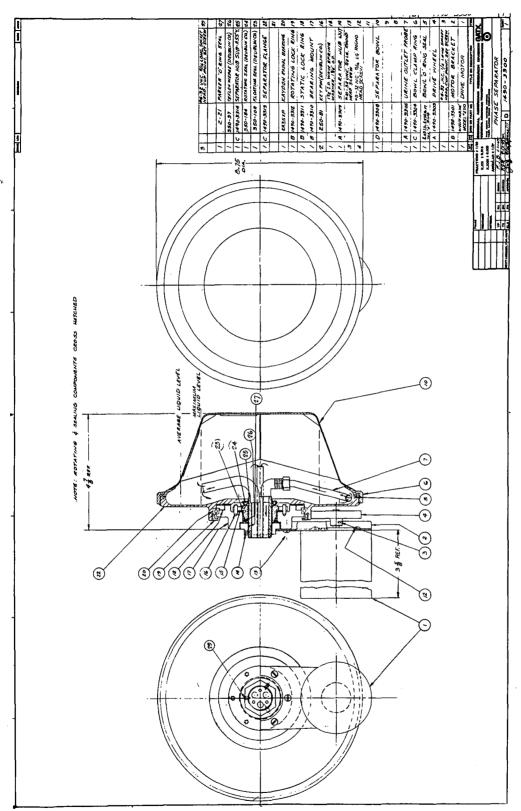
Phase Separator (Figure 6)

The phase separator configuration is a frustrum of a cone fabricated in two sections, a flange and a bowl, joined together with a clamp ring. These two parts are attached to a central stationary separator hub on a radial bearing so that the flange and bowl rotate. Rotation is accomplished by means of a 400 Hz motor mounted eccentrically on a bracket fastened to the stationary separator hub. Attached to the output shaft of the motor is a rubber wheel which tangentially drives a separator flange. A stud controls the contact pressure between the drive wheel and separator flange. Sealing between the stationary and rotating parts is provided by a light spring loaded face seal.

The stationary separator hub contains three tubes for access to the interior of the bowl assembly. These tubes are a urine and air inlet probe, an air outlet probe, and a urine outlet probe. The air outlet tube is on the geometric center of the assembly so that only the gaseous by-products of the urine are picked up. Both the inlet probe and urine outlet probe are turned approximately 105° from the centrifuge axis. The urine outlet probe is turned at the end so that the tube acts as a tangential scoop in the fluid. The inlet tube end extends radially and terminates at approximately two thirds of the radius distance inside the bowl.







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FIGURE 6 Phase Separator

Attached to the inside of the bowl are four equally spaced fixed vanes mounted parallel to the rotation axis. These vanes act as impellers to drive urine droplets to the inside walls of the bowl during weightless operation. The drive motor operates from a 115/200-volt, 3-phase, 400-Hertz, 4-wire source.

Acid Pretreatment Reservoir (Figure 7)

The pretreatment reservoir is a cylindrical tank with a gas-pressure loaded diaphragm to contain and dispense the acid pretreatment liquid. The 4:1:4 sulfuric acid-chromium trioxide-water mixture is stored in an acid resistant reservoir. The housing and lines contacting the acid are Teflon; the rolling diaphragm material is Hypalon. Acid is dispensed from the reservoir by gas pressure on the rear side of the rolling diaphragm. The reservoir capacity is 3 liters and overall dimensions are 8 inches in dia. by 13 inches long; a calibrated quantity indicator protrudes an additional 7 inches out of the rear end when the reservoir is full.

Acid Metering Valve

The pretreatment metering valve (Figure 5) is the same as the disinfectant injection valve.

Blower

Pneumatic transport in the urine collection subsystem is provided by a small diaphragm blower. The brushless d-c motor and blower assembly is a Brailsford & Co. Model TD-2A. The blower is mounted inside a sealed enclosure with an inert gas for compatibility with operating in a pure oxygen environment. The sealed enclosure contains removable plugs for periodic purging or resupply of the inert gas. The motor operates from a 28-volt DC source.

FIGURE 7 Acid Pretreatment Reservoir

Particulate Filter

A Pall Corporation Petro Sorb Ultipor .9 filter element is located in the phase separator line to the blower diaphragm. The filter element is supported within an anodized aluminum housing. The filter absorbs odors and removes particulates down to 0.08 microns in diameter.

Waste Holding Tank (Figure 8)

The waste holding tank is constructed of two hemispherical spinnings which are joined to a mounting rim at the tank's equator. One spinning has a neck and opening for inserting and securing a bladder; the opposite hemisphere incorporates four bosses for filling or removing liquid.

Waste water is admitted and stored between the tank wall and the bladder. The inside of the bladder is vented to cabin atmosphere via a porous plug.

The tank outer shell is stainless steel, the bladder is silicone rubber, and the bladder plug and retaining nut are acrylic plastic material.

Feed Control

The urine feed control is comprised of a metering accumulator (Figure 9) and a three-way solenoid valve. The connections to the valve are:

Port Connection

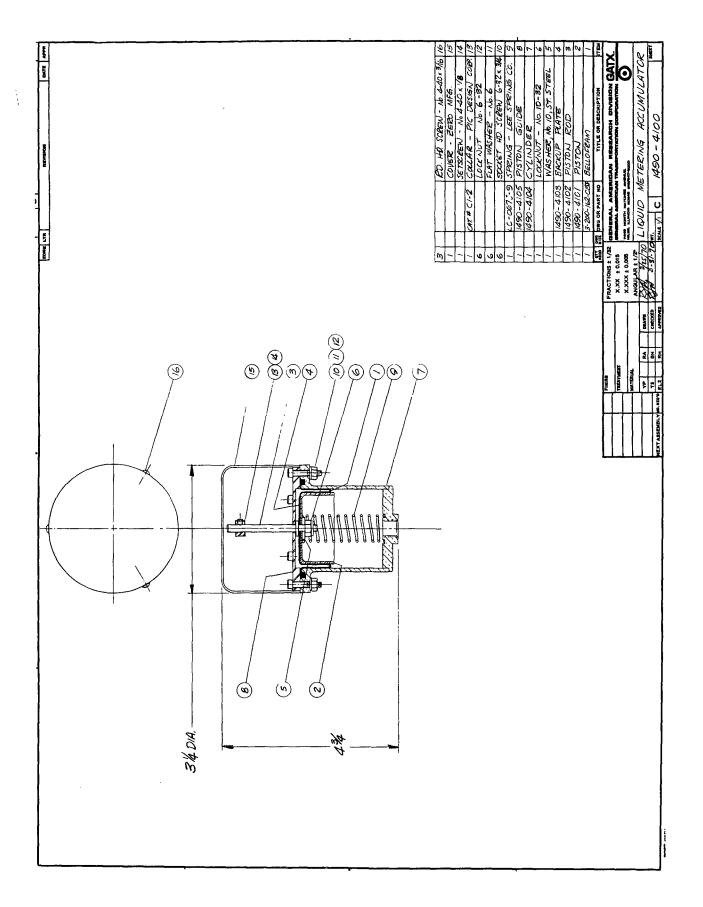
Normal Open Waste Holding Tank

Common Metering Accumulator

Normal Closed Evaporator

The feed control utilizes the pressure differential between the waste holding tank at ambient pressure, and the evaporator, at distillation pressure, to transfer a controlled amount of feed from the tank to the

FIGURE 8 Waste Holding Tank



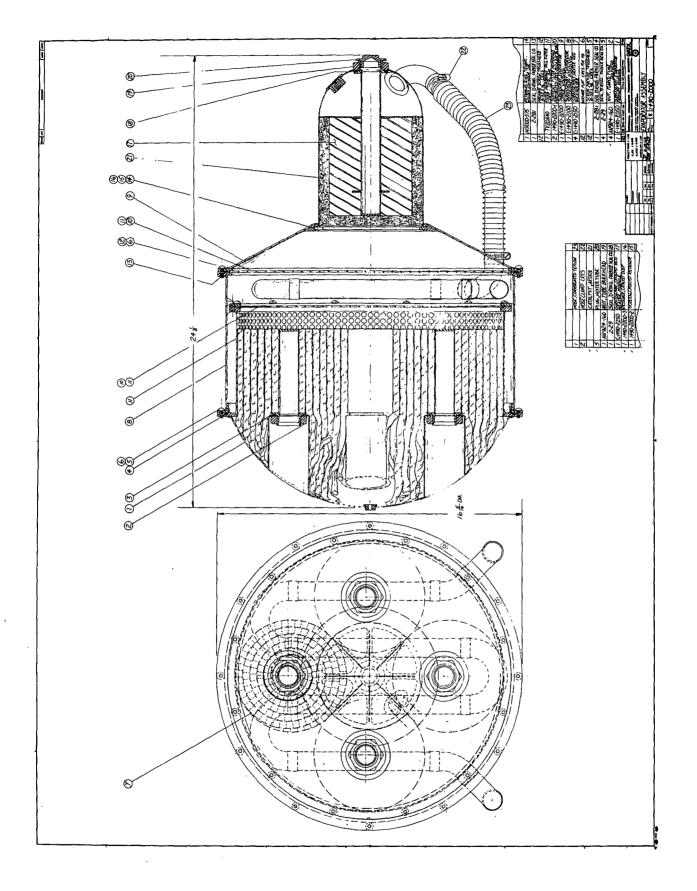
evaporator. When the three-way solenoid valve is actuated, the metering accumulator piston is forced down to empty the chamber and to compress a spring. When the solenoid valve is released to its normal position, the spring returns the piston to its stop and fills the accumulator with an amount of liquid equivalent to that fed to the evaporator.

Urine flow through the valve contacts only Teflon parts; the solenoid plunger is isolated from the liquid by a Teflon diaphragm. The three-way valve is manufactured by Fluorocarbon Co.

Evaporator Assembly (Figure 10)

The evaporator assembly includes the vacuum distillation unit, microporous filters, catalytic oxidation unit, and recuperator. The outer shell is fabricated of stainless steel spinnings, formed sheet and machined flanges into three weldment sections which are assembled to form the necessary vacuum vessel. The sections are joined via bolted flanges and O-ring face seals.

Four heat exchanger and wick modules are mounted within the lower cylindrical section. Each module is approximately five inches in diameter and 4-1/2 inches high. Strips of polypropylene felt are placed between radial fins around the heaters; the felt wick transports liquid from the lower hemispherical reservoir to the heat exchange surface and accumulates the residue from distillation. Each heat exchanger is capable of accepting a heating cartridge l-inch in diameter and 4-inches long. The heater may be a radioisotope heat source or an electric heater. The heat exchanger and wick modules are enclosed on the top and sides by a microporous filter, Pall TV20A40 material.



A cross-flow recuperator is located immediately above the upper face of the filter. It consists of 3-1/2 feet of 1-inch thinwall stainless-steel tubing through which the hot vapor discharge from the catalytic oxidation unit passes; the hot vapor gives up heat to the vapor outside the tubing coming from the evaporator distillation unit. The bleed gas required for the catalytic oxidation unit also enters immediately above the filter.

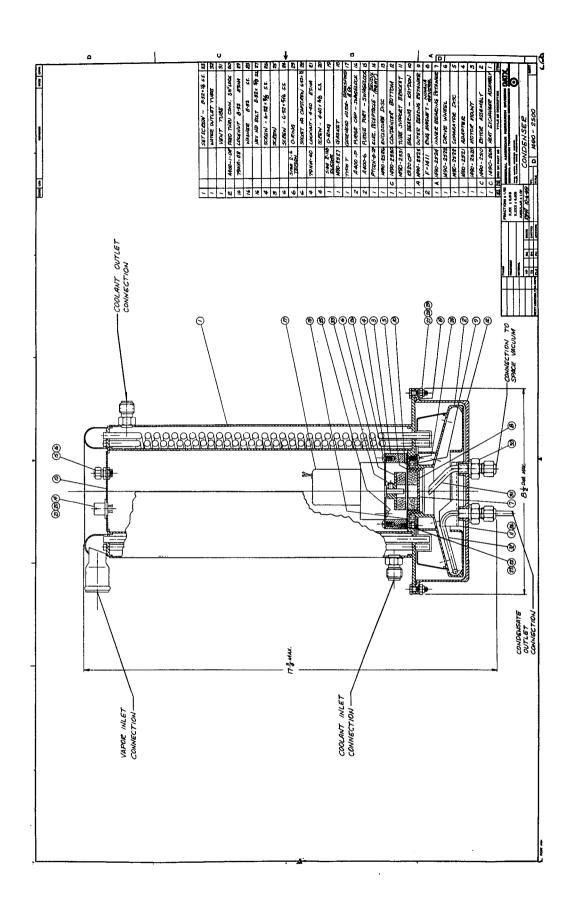
A second flat membrane filter, also of microporous Teflon material, is located between the top of the recuperator and the catalyst bed inlet. This serves as a final entrainment separator and as an inlet plenum for the catalyst bed.

A catalytic oxidation unit 5-1/2 inches in diameter and 6-inches high contains 4-1/2 lb. of ARDOX oxidation catalyst. A central heater housing in the catalyst contains a heating cartridge 1-inch in diameter and 4-inches long. Eight radial fins running the length of the bed are welded to the central heater housing. The fins serve to evenly distribute the heat across the catalyst and flow area of the bed. A fiberglass filter at the catalyst bed exit retains catalyst fines, 200-mesh size and larger, and provides a discharge plenum for the exiting vapors.

Condenser (Figure 11)

The system condenser assembly condenses the product vapor and separates the product condensate from non-condensibles. Heat transfer is accomplished through thirty (30) tubes wound into a helix of approximately 2 turns. Each tube is 1/4-inch OD by 30 inches long with .015-inch walls. The set of thirty tubes is enclosed by an inner and an outer shell through which coolant circulates.

Water vapor from the distillation unit enters the inlet plenum and is distributed to the 30 tubes. Coolant supplied from an external source removes



the superheat and the heat of condensation from the process stream. Coolant flow is around the tubes and in a counterflow direction to vapor and condensate flow.

As condensed droplets are carried by vapor and non-condensible gases to the discharge end of a tube, a sphere of water is formed which clings to the tube end by surface tension. Radial vanes in a rotary separator rotate closely past the tube ends and break the surface tension of the water sphere to form small droplets. The droplets are moved from the end of the tubes and are transported to the rotating separator. Centrifugal action then drives the liquid to the walls. A stationary pitot tube at the outer periphery of the separator allows flow of the accumulated potable water to the outlet fitting of the condenser. A second pitot tube located at a slightly shorter radial distance from the center senses the pressure differential when the tube becomes filled with liquid. This pressure differential actuates a switch which in turn controls the on-off activity of the water pump which then empties the water from the separator. Non-condensible gases in the process stream exit from the system through a purge tube located on the centerline of rotation.

The condenser phase separator is driven by a brushless motor which operates from a 28-volt DC source. The motor is coupled to the rotary separator by a magnetic coupling across a non-magnetic wall which forms a part of the vacuum structure; this allows the motor to be located in the ambient pressure environment and the separator to be driven in a sterilized vacuum environment without dynamic face seals. The motor is enclosed within an inert gas envelope to be compatible with operation in a pure oxygen environment.

Liquid Level Switch (Figure 12)

A differential pressure switch is utilized as the liquid level control for the condenser phase-separator and water pump. The high pressure side is connected to sense the pressure produced by a stationary pitot tube in the phase separator. The low pressure side is connected to sense the pressure in the vacuum purge line.

The switch body and internal piston are constructed from Lexan to be compatible with steam sterilization. Rolling diaphragms of surgical grade silicone rubber are used as the main sensing diaphragm and as a static seal around the switch rod.

Water Pump (Figure 13)

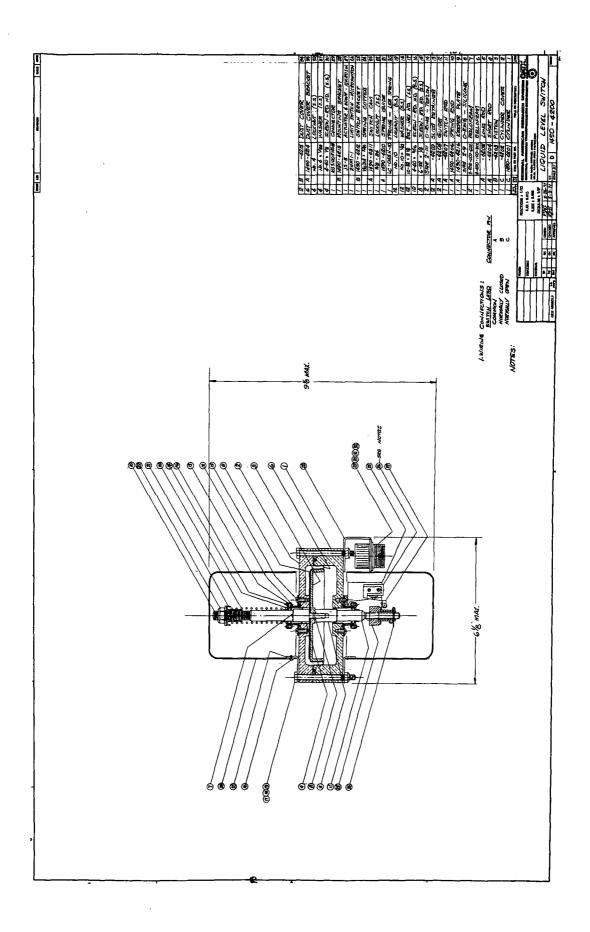
A review of positive displacement pump characteristics indicated that a diaphragm pump was best suited for this application. A dual head diaphragm pump requires power for pumping at 180° offset intervals of the crank. This allows the motor to be the smallest possible size.

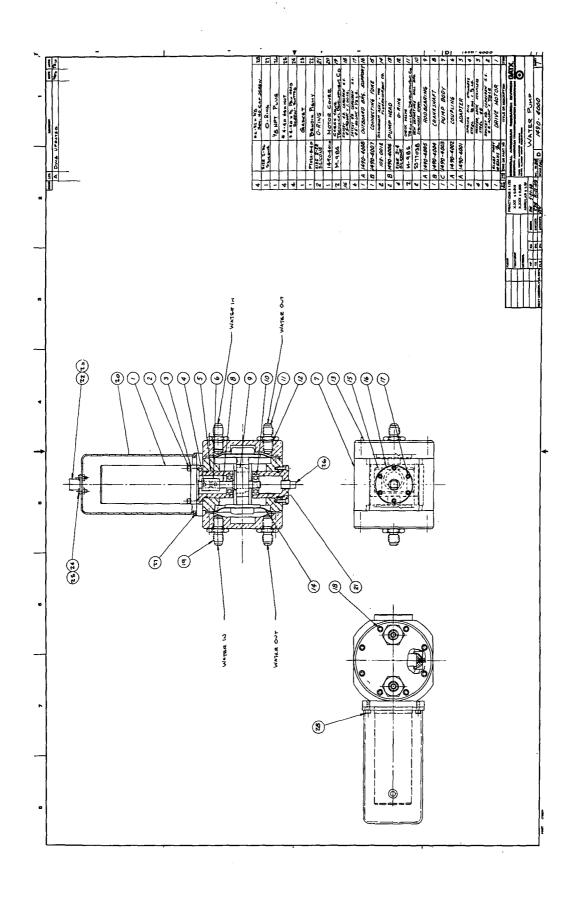
The pump heads are Teflon with Teflon diaphragms and are manufactured by the Fluorocarbon Company. The diaphragms are rated at over 20 psi pressure differential.

The check valves have an aluminum body and poppet with a Monel spring.

A very light poppet is mandatory for the inlet valve because this poppet must open at a maximum differential pressure of 1-inch of water. Both the intake and discharge check valves are manufactured by Technical Development Corporation.

The pump is driven by a gearhead motor at 13 rpm. The motor operates from a 115/200-volt, 3-phase, 400 Hertz, 4-wire source. The motor is blanketed in a inert gas enclosure to be compatible with operation in a pure oxygen environment.





The dual head pump was originally designed to provide equal flows by metering urine feed through one head and pumping an equal volume of product condensate through the opposing pump head. As dictated by shakedown testing, a separate feed control was required to more accurately meter the urine feed to the evaporator. The dual head pump was then converted to pump product condensate at each head.

Potable Water Tank

The potable water tank (Figure 8) is identical in size and fabrication method to the waste holding tank. For storage of water the metal parts were fabricated from aluminum to realize the lowest possible weight.

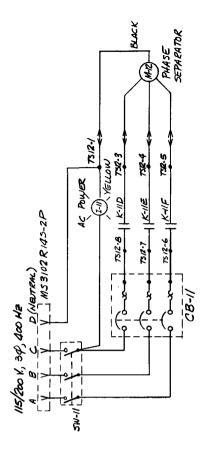
The mounting plug for the bladder incorporates a fitting for connection to a 4.0 psig regulated gas supply. This is required to dispense the water from the tank.

Electrical Controls

Electrical controls and displays are housed within and mounted in two control boxes located on the system frame. A urine collection and storage system control box (Figure 14) contains the switches, pilot lights, and circuit breakers or fuses for the urine collection phase separator and blower. Figure 15 is a schematic wiring diagram of the urine collection controls.

The water recovery system control box (Figure 16) contains switches, pilot lights, and circuit breakers or fuses for the condenser rotor, the water pump, and the feed control; individual warning lights indicate when process conditions are above the desired values for condenser vacuum,





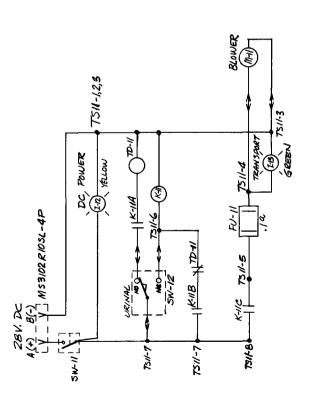


FIGURE 15 Urine Collection Wiring Diagram

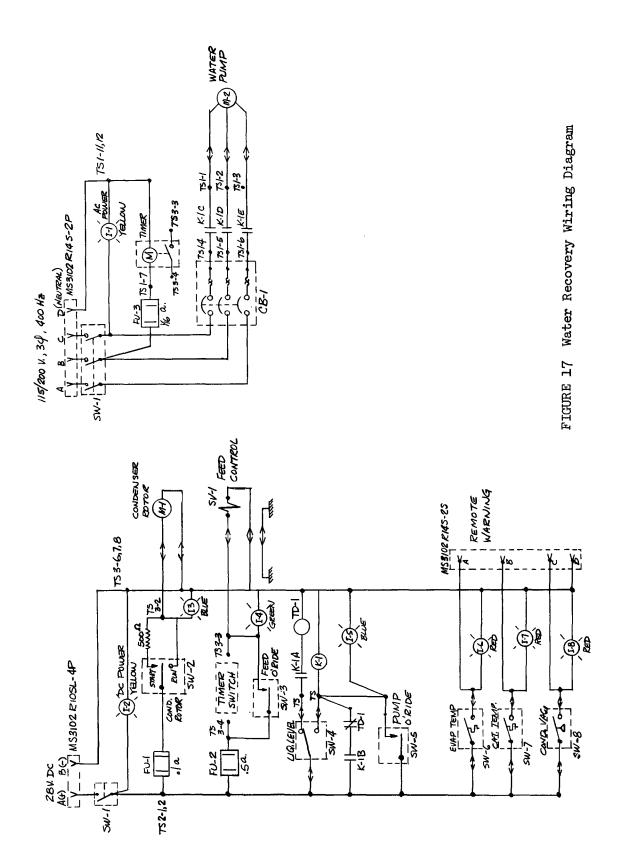
FIGURE 16 Water Recovery Control Box

catalyst temperature, or evaporator temperature. Figure 17 is a schematic wiring diagram of the water recovery system controls.

System Assembly

The general system (Figure 18) configuration is arranged for easy accessibility to individual components. Urine collection components are located in the lower section, water recovery components are located in the upper section and the large storage tanks for waste and water are located in the lower rear section.

The basic framework is extruded aluminum angle joined by riveted gussets and corner brackets. Components are bolted either directly to the angle frame or to intermediate brackets of formed aluminum sheet.



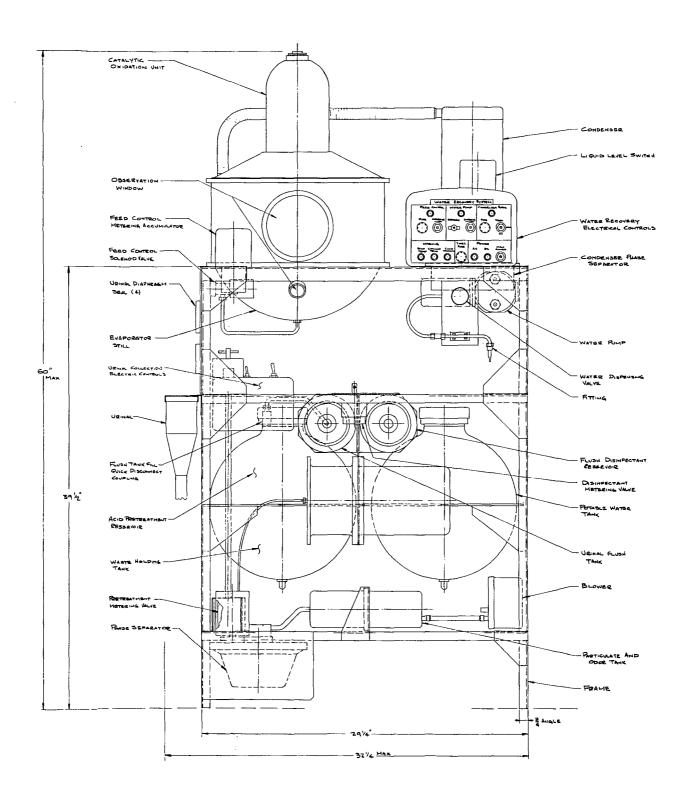


FIGURE 18 Water Reclamation System Assembly

SECTION III

OPERATION AND TESTING

The Water Reclamation System was tested in two phases: (a) laboratory glassware evaporator verification tests, and (b) hardware water recovery system performance tests. In phase (a) the laboratory evaporator was verification tested to show specific component operating characteristics; in phase (b) a full-scale evaporator assembly, with a catalytic oxidation unit and auxiliary system components, was tested for a thirty-day period to demonstrate system operating and performance characteristics.

The basic concepts tested and the objectives of testing were:

- 1. Distillation of urine at reduced pressures and temperatures, in the range of 25 to 40 mm Hg, and 80 to 120 F, respectively. Objectives were to show rates of water recovery and the effects of pressure and temperature on generation of gases and vapors other than water during distillation.
- 2. Separation of distilled vapor from the boiling liquid by means of a hydrophobic membrane material. Objectives were to show the ability and efficiency of the membrane to constrain liquid and allow vapor to pass through, and to show the durability of the membrane when in contact with urine liquid for prolonged periods, while the urine solids concentration increased with time.
- 3. Transfer of heat from the heating source to the liquid being distilled. Objectives were to show the effectiveness of the heat transfer system (heating elements and wicks), and rate of heat transferred, surface temperature excursions experienced, and

- effects of urine solids build-up on heat transfer at the boiling liquid-vapor interface.
- 4. Flow control of urine feed and condensate removal. Objectives were to show feasibility of simultaneous pumping in of feed urine, and pumping out of condensate with one double-ended pump, to automatically maintain a fixed amount of liquid within the evaporator system.
- 5. Conversion of trace organic compounds in generated water vapor by catalytic oxidation. Objectives were to show that organics in the water vapor can be catalytically oxidized to CO₂ and water at relatively low temperatures, 250 to 300F, by reaction with a small amount of oxygen bled into the generated water vapor.

DESIGN VERIFICATION TESTS

Testing of basic concepts commenced during the design phase and specific verification tests were conducted. A glassware distillation unit was constructed from a 16-inch diameter by 7-inch high plexiglas cylinder with flat end-plate to evaluate the vapor-liquid separation membrane and liquid retention wick materials. The same unit was used for bread board testing of the urine feed-condensate removal control unit. Figure 19 shows the verification test unit.

Membrane Tests

The membrane tests were started with Pall TV2OA40 membrane material and 1-inch glass fiber media as the pre-filter. The longest continuous run was 233 hours with an average of .62#/hr of water produced from pretreated urine. The test was terminated after 233 hours due to leakage of urine liquor droplets through the membrane. This represented the most successful of four runs in which new membranes were installed for each run; also, one membrane assembly leaked excessively during its initial static checkout with water and had to be

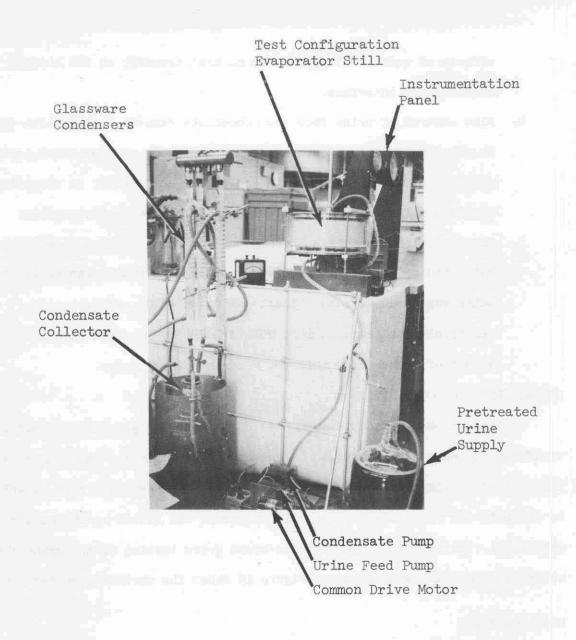


Figure 19 DESIGN VERIFICATION GLASSWARE SYSTEM

discarded. In view of these results and the fragility of membrane materials, it was decided to investigate configurations in which the membrane would serve as a vapor filter but in which liquid would be precluded from contacting the membrane by constraining the liquid in a wick.

Wick Tests

The distillation test unit was re-assembled with Dacron felt wicking material wrapped around the heating element housing. A 1-inch void space was maintained between the top of the wick assembly and the membrane to prevent the body of liquid from contacting the membrane. Dacron felt was used for the wick in this test because it exhibited the best wicking properties of materials which were immediately available in sufficient quantities for use in the test unit. During the continuous run of 245 hours, 99 liters of water were recovered from 103 liters of urine. The test was terminated when solids entrained by vapor and deposited on the membrane were extruded through the primary membrane.

These tests, conducted on the research distillation unit, indicated that using the microporous membrane as a vapor-liquid separator for an extended period of time is not feasible for periods over 1 week of continuous operation. Also, operation with liquid kept away from the membrane by wicks around the heaters showed promise, although this particular configuration was limited by the amount of heat transfer area in the body of the wicks. The results of the design verification tests using the membrane as a vapor-liquid separator are summarized in Table I.

After these tests were completed the vacuum distillation section of the evaporator assembly was redesigned. A new configuration was designed on the basis of the results of the tests discussed above and on tests which were concurrently being conducted for AMRL and Mound Laboratories.

TABLE I
PERORMANCE OF MEMBRANE AS VAPOR-LIQUID SEPARATOR

Date	Test Duration Hours	Urine Feed Liters	Condensate Collected Liters	Remarks
6/30/69 to 7/09/69	30	14.1	11.0	Shutdown - Leaks through lower membrane. Liquid contacting membrane
7/14 to 7/16	27	10.7		Shutdown - leaks Liquid contacting membrane
7/21 to 7/26	48		~ -	Tubing Pump failed resulting in urine flooding evaporator & rupturing membrane.
7/29 to 8/06	233	70.3	66 . 5	Shutdown - 3/4 in. of liquor between membrane & plexiglass cylinder.
week of 8/11	leaked ex No liquid	cessively when I in direct cont I around heaters	tested with wa act with membr	because previous ones ter against membrane. ane. Dacron wick id space between wick
8/18 to 9/02	345	103.15	99	Shutdown - Liq. & solid residue from vapor entrainment being extruded through membrane.

Wick and Finned Heater Tests

The new still section consisted of a module with heat-transfer fins extending from the heater housing surface to provide better heat transfer into the wicking material. The fins were stainless steel, attached by brazing to the stainless steel heater housing. The new configuration, with extended heating fins and wicks was assembled and lab tested in a glassware evaporator 10-inches in diameter and 7-inches high, with a flat top, and a hemispherical bottom. Thermocouple probes were placed at approximately midheight of the wicks at radial distances of 3/4, 1-1/8 and 1-3/4 inches from the cylindrical heater housing. Pretreated urine (4.5 Liter) was fed into the hemispherical bottom of the evaporator; the level of the liquid was maintained initially at the bottom of the heat exchanger, but the wicks immediately drew the liquid up entirely around the 4-1/2 inch high heater. Heater power was set at 110 watts. The module evaporated 20 liters in 122 hours at pressures of 22 to 28 mm Hg during which time the temperature at the inner probe, 3/4 inches from the heater, rose from 80 to 140F; The maximum temperature was limited by a control to 140°F, and the heater was on approximately 75% of the time, as evidenced by a drop in recovery rate from 4 to 3 liters/day. The boiling point at 22 to 28 mm Hg rose as the urine solids concentration increased from approximately 4% initially to the 30% to 35% level. However, the temperatures observed were still considerably above the increased boiling point range of 82 to 80F for these pressures and urine concentrations, indicating that overheating was occuring and that the wicking material was not transporting urine rapidly enough to the heating surface.

The wicking rate for the 4-1/2 inch vertical height of the wicks and heater assembly decreases when the wick becomes fouled with urine solids. When the rate of urine transport within the wick decreases, the amount of evaporation resultingly decreases. The amount of heat input is constant, since the heater is on, and any heat above that needed to evaporate urine will enter the surroundings, namely the only partially saturated wick. The temperature of the wick will continue to rise until it approaches the control setting of 140 F. At this level some urea breakdown can occur with evolution of NH₂.

To provide for more nearly complete saturation of the wick and better heat transfer during the remainder of the test, the liquid level in the wick was raised to approximately 1/2 the height of the heater elements. This caused the wick temperature again to be lowered; the radial temperature distribution, after 55 liters total of urine input, was 95, 94 and 91°F. The verification test was stopped at this time since the capability to sustain evaporation at desired temperature levels along with the ability of the wick to sustain urine transport, appeared to be satisfactorily demonstrated. The 95 to 91 F temperatures in the wick, at a pressure of 25 to 30 Hg, corresponded to an apparent solids concentration of 60%; this concentration was more than would be required to operate the evaporator to completion for the intended 40 day use cycle.

The tests did indicate that the urine level, and the height of wick activity for urine transport and evaporation differed from the original design values.

SHAKEDOWN TESTS

After completing the verification tests, a full-scale hardware evaporator was fabricated along with support hardware components. The evaporator assembly, condenser and dual urine metering and potable water pump were shakedown tested, both as individual components and then as a system, to demonstrate system performance and ability to meet operational requirements. Approximately 72 liters of tap water were processed at or in excess of the four-man evaporation and condensation rates during initial shakedown tests. The total time elapsed during these tests was approximately 15 days.

The major system components functioned properly with the following exceptions:

(1) The urine feed control malfunctioned after metering 42 liters of tap water feed. The failure was traced to a check valve in the metering pump. The failed valve allowed the evaporator to siphon water from the feed tank. Since considerable trouble was experienced with this valve during the pump's checkout test, it was decided to use a solenoid valve for positive shutoff in the feed line. The urine feed control was altered to utilize a 3-way solenoid valve and a small spring-loaded diaphragm accumulator instead of the original metering pump. (The new feed mechanism is described in Section II, Major Components.) The 3-way solenoid valve actuates at the same time the water pump operates and the volume in the metering accumulator was adjusted to feed an amount of urine into the evaporator equal to the amount of condensate transferred to storage by the diaphragm pump.

- (2) The condenser operated to transport non-condensibles to the condensate storage tank on several occasions. At certain times the volume of condensate contained within the rotating phase separator in the condenser was not sufficient to insure that the liquid pickup probe was always running in liquid. If the amount of liquid present was low and the timer actuated the water pump, non-condensibles would be admitted to the pump inlet, causing the pump to lose its prime. An expansion reservoir was thus required between the condenser phase separator and the water pump. The expansion reservoir insures that water will always be available at the pump inlet.
- (3) On several occasions the phase separator in the condenser failed to start rotating although the drive motor was operating. When the full 28 volt dc voltage was applied instantaneously the magnetic coupling disconnected from the rotor due to the rotor's inertia. To overcome this inertia a motor starting switch was required for the phase separator motor. By starting at a lower voltage and lower motor speed, the rotor was moved from its rest position before the full power for high motor speed was applied.

CHECKOUT TESTS

The water recovery components consisting of an evaporator, the condenser, water pump, feed control, and potable water tank were connected with the modifications indicated by the Shakedown Tests. The system was then tested to verify its vacuum tightness, and the potable water components (from the outlet of the ARDOX catalyst bed to the potable water tank outlet) were sterilized by admitting 212F steam through the components for 4 hours.

The 5-day checkout test was started on April 15 and continued through April 20, 1970. The modified urine feed control, the condenser expansion reservoir and the condenser phase separator all functioned properly. The system performed at the required four-man urine and waste water distillation and condensation rate with 400-watt input to the evaporator still. The evaporator operated with wick temperatures between 83 and 106 F at pressures from 15 to 37 mm Hg, with coolant running at 40 to 60 F. The product condensate was found to be "essentially sterile" with the MPN < 2.2/100 ml for viable organisms.

The system was shutdown on April 20 because the evaporator had overheated while operating unattended overnight. The overheat failure was caused
by a freezeup in the vacuum system's dry-ice acetone trap. The loss of
vacuum, in turn, caused the urine metering accumulator to underfeed and
the evaporator to run dry.

The system was disassembled and the evaporator heat exchanger modules were cleaned in a warm potassium hyroxide solution and an ultrasonic cleaner. After re-assembly, the system was vacuum leak tested and the water loop sterilized with 212F steam for 4 hours. The systems controls were modified to incorporate a warning light which indicates (1) loss of vacuum at the condenser purge line, or (2) overtemperature at the evaporator

isotope housing. Since the system operated continuously and was unattended for the major part of a day, the warning control was also connected to shut off the evaporator heaters and the feed control and water pump. The warning system was connected so that it required manually restarting the heaters and liquid control, thus precluding a possible condition in which the system could cycle on again and go unnoticed. Shutting off the electric heaters simulates the physical removal of the isotopes from the evaporator.

An electric heating tape was installed on the inlet line to the dryice acetone trap to aid in preventing the trap line from freezing up.

Also, a constant voltage transformer was placed in the electrical heater
line to more closely simulate the constant heat provided by isotopes.

The check-out tests indicated overall satisfactory system operation and the system was prepared for the 30-day evaluation performance test.

PERFORMANCE TESTS

Dry Ice Trap Water

To evaluate performance under simulated input rates, the water recovery subsystem and the potable water storage subsystem were subjected to a 30-day performance test. Electric heaters were used to simulate the heat supplied by radioisotopes. The test was conducted in GARD's research laboratory at normal atmospheric conditions.

Instrumentation and Monitoring

The operating conditions during testing were measured and recorded by the instruments listed below:

Measurement	Instrument
Process Temperatures	Honeywell multipoint recorder (0-300°F range) and T/C
Heater Housing Temperature	Indicating Temp. Controller & T/C
Evaporator Pressure	Honeywell Aneroid Pen Recorder (0-100 mm Hg Absolute)
Condenser Pressure	Absolute Mercury Manometer
Evap. Htr Power (simulated isotopes)	Sangamo Electric Watthour Meter (integrated) and Simpson (0-750) Wattmeter (instantaneous)
Catalyst Heater Power (simulated isotope)	Simpson Wattmeter (0-300 w)
Condensate Pump Power (400 Hz)	Weston Polyphase Wattmeter
Condenser Motor Power (28 vdc)	Weston DC Voltmeter and Weston DC Ammeter
Catalyst Bleed Gas Flow	Brooks Rotameter
Urine Input Water Input Water Produced Dry Ice Trap Water	Graduated Beaker and/or Balance-lab scale

Daily measurements were made and recorded for each of the above parameters. In addition, the process temperatures and the evaporator pressure were continuously recorded by the multipoint recorder and the aneroid pen recorder, respectively.

All urine processed was collected from male in-plant personnel with no control of the donor's diet. The collection was made in approximately 9 liter quantities during the normal working days of the week. Within 3 hours of the time when the collector was set out, the collection jug was returned to the laboratory and the urine was dosed with 4 cc/liter (6 cc/liter during some parts of test) of 4:1:4 H₂SO₄, CrO₃, H₂) pretreatment acid and 4 drops/liter of Dow Corning H-10 antifoam. This was usually accomplished during the afternoon of each weekly work day.

Simulated respiration and perspiration condensate (tap water) was added to the waste holding tank in quantities of approximately 13-1/2 #/day during the week and 34 #/day on weekends. Urine was added at 20-1/2 #/day only during weekdays since fresh urine was not available on weekends.

In-House Water Quality Analyses Procedures

The water quality monitoring program consisted of the following analyses and measurements: (1) Total Carbon (TC), (2) ammonia, (3) urea, (4) pH, (5) conductivity, (6) appearance, and (7) viable bacterial density. The analytical procedures for the above analyses are described in the following discussion.

Total Carbon analysis was performed with a Beckman, combustion type Total Carbon Analyzer. The analysis is performed by injecting a portion of the product water into the instrument's combustion tube which is maintained at 1600C and is continuously swept with a stream of pure oxygen. All carbonaceous material is converted to carbon dioxide in this tube. The CO₂

content of the oxygen stream venting from the combustion tube is analyzed by a special infrared dectector. The carbon content of the sample is obtained by comparing the observed instrument response to that obtained from acetic acid standards.

Ammonia and urea analyses were performed by colorimentry. The basic procedure for both substances was to treat a sample of the product water with a reagent composed of phenol, nitroprusside, and hypochlorite. The reagent, in the presence of ammonia, produces a blue color measureable at 630 mu. For urea analysis, urease enzyme, which converts urea to NH₃ and CO₂, is added prior to adding the above reagent.

Conductivity and pH were measured with standard laboratory instruments.

A Beckman expanded scale pH meter and an Industrial Instrument Conductivity bridge with calibrated conductivity probes were used, respectively, for measuring pH and conductivity.

Appearance was determined on the basis of subjective observation. Color was measured with a standard chloroplatinate color comparator.

The viable bacterial density was determined using a modified Most

Probable Number (MPN) procedure. The modifications consisted of using
thioglycolate broth in capped tubes instead of lacose broth fermentation
tubes. In the modified procedures, five 10 ml portions of the undiluted
sample, five 10-ml portions of a 1 to 10 dilution, and five 10 ml portions
of 1 to 100 dilution were innoculated into thioglycolate tubes. The tubes
were then incubated at 35 C for 48 hours. Gas formation within the broth was
considered a positive indication of coliform organisms. Viable bacterial
density of the product water was determined from standard MPN tables on the

basis of the number of positive tubes in each dilution level. The major advantage of the procedure lies in the fact that tests for the presence of both aerobic and anaerobic organisms are combined into a single determination.

Operation Data and Performance Results

The performance test on the main water recovery system components was conducted for 33 consecutive days from April 29 through June 1, 1970. The actual cumulative operating time was 756 hours, while operational interruption time was 37.5 hours, as listed in Table II. Interruption time is defined as that time when the system vacuum and/or evaporator thermal input was shutoff. Of the 37.5 hours total interruption time, 29.5 hours were caused by the test support equipment, namely a vacuum pump and an external liquid level sensor, and 8 hours were required for corrective action for the water recovery system to replace a corrugated Teflon hose which developed a leak. No other shutdowns were experienced. Minor repairs not resulting in interruption of system operation were required to clean a sticking check valve on the water pump and to replace a ruptured diaphragm on the water pump.

System Performance

The overall performance of the system is shown in Table II and III. Evaporation of the urine and waste water mixture took place at an initial temperature of 82 F. The liquid level in the evaporator ranged between two and three inches high in the cylindrical section of the still. The hemisphere below the cylinder was also filled at all times. The generated vapor temperature was measured just below the separation membrane about 2 to 3 inches above the vapor-liquid interface.

TABLE II - WRS URINE FEED & PRODUCT WATER RECOVERY BALANCE DATA

REMARKS	Startup @ 0730	level sensor used for evap.	safety backup.			Replaced corrugated Terlon hose.		Water line opened to	CTEMI CHECK VALVE	Caused by loss of vacuum	which shuts down system;	Tuse blown op trap line heater.	- Water line opened to	replace pump diaphragm.																				- 1	st: 25 watts 30 cc/min (STP)		
INTERRUPTION TIME (hr)	2	7 -5.5	<i>-</i>			2 8 0 ×						S 16.0 ~																_+							Thermal input to ARDOX Catalyst:	4 drops antifoam per liter urine	thru 5-31
VACUUM LINE FREEZE TRAP (1b)	п.	.31	₹8°.	*:	11.11	1.26	1,61	1,88	2,15	2.45	2.72	2.94	3.20	3.53	3.81	4.10	4.38	4.63	4.93	5.24	5.56	5.89	6.39	6.89	7.24	7.57	8	8,43	8.93	9.32	09.6	9,92	10.87	17.41	Thermal inp	4 drops ant:	added, 5-7
RECOVERED WATER (1b)	10.5	29.2	62.0	91.4	121.4	148.2	180.4	213.7	243.0	277.3	303.6	336-1	352.2	385.0	418.4	0.644	481.5	506.8	542.4	67.65	608.5	641.7	6.179	705.8	728.8	760.4	795.2	826.7	859.8	892.7	922.6	951.5	983.5	1013.3			- 5/31
TOTAL INFUT (A)	58.1	87.1	116.1	149.1	177.3	199.6	228.7	277.5	299.7	342.5	364.5	397.5	420.5	451.6	490,1	525.6	9.095	582.6	615.6	650.6	677.4	719.0	758.9	796.8	818.8	840.8	887.9	919.3	953.3	986.5	1015.1	1045.9	1067.9	1067.9 (B)			c/liter, 5/12
URINE INPUT (A)(D) (1b)	18.5	47.5	74.3	74.3	74.3	6.%	126.0	159.6	170.8	180.4	180.4	180.4	203.4	223.5	242,2	264.5	281.9	281.9	281.9	297.1	323.9	339.1	361.4	388.3	388.3	388.3	411.2	425.0	445.8	461.4	4.194	1,161.4	761.4	461.4	(၁)		4/29 - 5/11; 60
EVAPORATOR THERMAL INPUT (Kw Hr)	0.4	9.3	19.7	27.6	37.2	45.7	55.6	65.6	75.0	85.1	93.5	104.1	109.5	119.5	129.7	139.3	150.3	157.3	168.3	179.8	189.3	199.5	209.1	219.9	227.6	238.0	249.6		269.1	279.1	288.3	297.5	307.7	316.2	holding tank.	end of test.	e: 4cc/liter,
OPERATION (HRS)	9.5	18.5	1.1	63.1	86.5	107.5	131.2	155.5	178.5	202.7	222.8	248.2	259.3	283.0	307.0	330.0	354.5	373.0	399.5	427.2	450.8	474.8	1,98.0	524.0	542.8	568.0	595.8	617.0	642.8	6,999	888.8	0.17	735.0	0.957	a)	y at end of test.	Acid pretreatment added to urine: 4cc/liter, 4/29 - 5/11; 6cc/liter, 5/12
TIME OF RECORDING	1700	1530	1740	1220	11.35	1630	1615	1630	1530	1540	 02 11	1315	1620	1600	1645	1530	1600	1030	1300	1640	1545	1545	1500	1700	1145	1300	71645	1400	1545	1550	1350	1200	1200	006	Includes amount in waste	Waste tank empty at end	Acid pretreatme
Date	4-20	1-30	1 7	5-2	5-3	4-5	, , , , ,	9. 14	12	- 2	6.4	5-10	5-11	5-12	. F	5-14	5-15	5-16	5-17	5-18	: - 61-7	5-20	्ट <u>-</u> र	5-25	5-23	5-24	5-25	5-26	5-27	5-28	5-29	5-30	£-3	6-1	(A)	(m)	(<u>a</u>

TABLE III - WRS PRESSURE & TEMPERATURE PERFORMANCE DATA

																						,				4.5			STATE OF	GH42	-			455
Cond. Coolant Out	66°F	1 9	61	09	09	62	61	. 65	61	1 9	61	79	65	19	61	61	61	61	62	61	61	62	61	29	62	29	65	61	62	63	69	65	70	63
Cond. Coolant In	55°F	54	50	50	51	51	50	64	51	52	50	55	1 2	20	20	50	.05	50	51	50	20	52	51	52	64	ſζ	53	64	50	50	58	55	63	50
Cond. Vapor Inlet	85°F	80	78	92	92	62	80	81	82	82	78	83	83	81	80	81	81	81	81	81	42	8.	85	98	88	87	87	85	81	81	83	24	82	75
Cat. Vapor Outlet	200°F	205	212	214	215	225	210	219	220	220	215	215	215	212	210	216	216	219	219	219	217	220	219	222	221	220	222	220	218	219	221	218	220	21.3
cat. HTR HSG	240°F	254	246	246	546	246	240	545	546	546	248	238	546	242	242	246	246	248	245	250	246	546	546	250	250	245	242	545	250	546	546	546	242	242
Cat. Vapor Inlet	115°F	115	123	121	120	118	911	971	125	130	123	125	011	110	† TT	120	120	121	011	115	120	711	115	121	121	122	122	977	7	113	115	† 17.7	17	100
Evap. Vapor	84°F	82	82	81	80	8	85	98	88	68	89	8	89	91	89	%	8	8	101	103	103	011	108	109	108	105	107	106	706	105	108	108	112	11.5
Evap. Liquid	84°F	82	82	82	85	88	87	87	96	91	35	95	89	93	멍	66	66	100	100	106	106	111	011	112	777	112	113	011	971	119	120	971	120	126
Evap. HTR HSG	130°F	06	011	93	100	148	148	132	118	120	120	66	66	120	8	120	125	8	700	120	011	100	132	120	120	120	711	11.7	110	110	011	120	110	011
Cond. Press. (mmHg)	18	15	77	7.7	47	7,7	17	15	15	77	1.5	15	14	17¢	17	14	14	7,7	14	_ት ር	15	50	91	21	19	18	18	18	97	16	15	13	97	14
Evap. Press. (mmHg)	25	55	12	50	19	55	22	21	22	21	22	13	23	27	21	21	23	21	50	23	50	27	54	59	28	58	28	28	₹	27	25	21	32	37
Date	1-29	4-30	7.	5-5	5-3	7-4	5-5	2-6	2-5	5-8	5-9	5-10	11-5	5-12	5-13	5-14	5-15	5-16	5-17	5-18	5-19	5-20	5-21	2-52	5-23	5-24	5-25	5-26	5-27	5-28	5-29	5-30	5-31	6-1

For the initialy dilute solution, approximately 3% total solids, the saturated vapor pressure is about 27 mm Hg. The observed distillation pressure was measured at 21 to 22 mm Hg during the first 20 days of testing. This pressure was measured above the vapor-liquid separation membrane over the boiling compartment, rather than directly in the boiling compartment. The apparent pressure drop across the membrane was in the range of 4 to 6 mm Hg, so that the recorded evaporator pressure is thus lower than the actual value at the liquid-vapor evaporation interface.

As evaporation continued and the solids concentration increased the boiling point rose to 106 F at the end of the 20th day, while the pressure remained relatively constant. From the 20th day on to the completion of the test both evaporation temperature and measured pressure rose even further. Temperature rise was attributed to increasing solids concentration, while the apparent rise in pressure may actually have been due to a decrease in pressure drop across the vapor-liquid separation membrane. The membrane, after exposure to wetting by entrained droplets for extended periods, appears to transport vapor more readily than in its initial dry condition with a subsequent decrease in pressure drop.

The rate of heat transfer from the evaporator heater through the wick and heat transfer surface to the evaporating liquid, appeared satisfactory since the heater housing remained essentially in the range between 100 and 120 F, while the liquid remained below 125 F for the entire test. No excursions to the 140 F level were experienced.

However, it was necessary to control the feed to the evaporator manually to maintain the desired liquid level of 2 to 3 inches above the bottom of the heater housing in the still. In this manner the wick material was

kept saturated, and the proper distillation rate was maintained.

The recuperator above the membranes recovering heat from the gases leaving the catalytic oxidation unit was partially effective during the earlier part of the test period but appeared to lose its efficiency during the latter part of testing. Its overall effectiveness was not significant in conserving on the total energy input to the system.

The condenser operated properly in converting vapor to condensate at approximately 15 mm Hg, corresponding to a condensation temperature of 64 F. This temperature closely approximated the temperature of the coolant leaving the condenser, indicating that the condenser was adequate to condense all of the vapor generated at the rated capacity of the water recovery system.

The liquid level sensing system in the liquid-vapor separator at the bottom of the condenser properly controlled the liquid transfer pump. The pump transported condensate liquid from the 15 mm Hg abs level up to 5 psig pressure for storage and dispensing as needed.

As shown in Table III cumulative feed to the system was:

Urine	461.4 lb
Tap Water	606.5 lb
Total Feed	1067.9 lb

Recovered water amounted to	1013.3 lb
Water lost in vacuum line freeze trap	11.4 lb
Liquid and solid residue in evaporator at end of test	34.9 lb

Comparing the measured total inputs and outputs and final residue, 8.3 lb of input remains unaccounted. Quantities of product gases from the catalytic oxidation process exiting the system were not monitored and could

contribute to this difference. In porportion to the total amount of water processed, the 8.3 lb represents less than 1% which is satisfactorily within measuring limits.

The resultant average daily system operating rates are:

Urine feed	14-1/2	lb/day
Total feed	34	lb/day
Recovered Water	32	lb/day
Loss in trap	1/3	lb/day

Product Water Quality

The quality of water produced is shown by analyses in Tables IV, V, and VI. The product water appeared to meet essentially all of the standards for potability designated for this system, with the exception of the subjective requirements for odor and taste. The product water in some instances had odors which were not specifically quantified and therefore not relatable to the requirement that they be unobjectionable. The same considerations applied to the taste of the water.

The amount of ammonia present in the product water was high by comparison to a generally desired level of 10 mg/l. The occasionally high ammonia levels in excess of 50 to 100 mg/l appear to be related to pre-treatment acidification of feed urine, and to the distillation temperature. A distillation temperature maximum of 100°F appears desirable to keep the ammonia level below the desired maximum. Occasional hot-spots in the heater and wicks appeared to generate excessive amounts of ammonia.

In-house water quality analyses are given in Table IV, while more complete analyses performed by Aerojet-General Corporation on samples which were collected and mailed on approximately every fourth day of operation are shown in Table V.

TABLE IV - WRS IN-HOUSE PRODUCT WATER QUALITY ANALYSES

SAMFLE COLLECTED FOR DETAILED ANALYSIS AND MAILED TO AEROJET-CENFRAL		·	· ·	No. 1		: :		No. 2				:	No. 3			No. 14			!	No. 5				No. 6					No. 7	::				No. 8
COLOR (Cobalt Units)	< 5	· ·	× ×	< 5	< >	< 5	\ \ \	< 5	< 5	. v.	< 5		< 5	< 5	< 5	< 5	< 2	< 5	< 5	< 5	5 >	< 5	< 5	< 5	< 5	< 5	< 5	\ \ \	< 5	< 5	< 5	\ \ \	< 5	< 5
UREA (<u>mg/1</u>)	< 2	∾ ∨	< 2	< 2	۵ ۷	2 >	01 V	2 V	8 >	< 2	∾ ∨	« V	α ∨	2 > .	۵ ۷	23 >	۷ ۷	8		< 2	< > >	2 >	۵ ۷	ر د د	< 2 >	< 2	< 2	α ∨	0 V	۷ ۷	2	a ′ ∨	۵ ۷	< 2
AMMONIA (mg/l)	0.5	1.1	7.	6.	.5	8.	1.6	22	%	180	88	r.	₹,	8.7	10.8	9.3	18	† 11	85	16	. 30	& ~	6.8	4.7	. 52	27	27	16	#	16	50	27	† 1	10.2
TOTAL CARBON (mg/l)	0	0	. 4	3	1	0	0	36	13	9.8	8.	9.8	0	0		0	. 80	13	. 6	1.6	, o	6	0	1.5	! _∞	1.5	10	ا به	0	0	10	0	0	0
CONDUCTIVITY	5000	5000	1 006	580	360	190	225	48	8	223	1777	700	132	04	84	39	53	150	02	89	. 89	69	17	26	. 20	ር	77	62	57	245	. 09	20	94	89
പ	2.2	2.3	2.9	3.1	3.5	3.7	3.7	0.6	6.6	2.6	4.6	9.4	8.8	0.6	2.6	8.9	9.1	7.6	9.3	0.6	1.6	0.6	6.3	8.9	9.1	2.6	2.6	8.9	0.6	7.9	0.6	8.9	0.6	8.3
Date	t-29	η - 30	 것 	5-2	5-3	5-4	5-5	2-6	52	5-8	5-9	5-10	5-11	5-15	5-13	5-14	5-15	5-16	5-17	5-18	5-19	5-20	5-21	5-22	5-23	5-24	5-25	5-26	5-27	5-28	5-29	5-30	5-31	6-1

	U.S. FUBLIC HEALTH SERV. POTABILITY LIMIT	5000	300	1000	50	1700 200	: ! : '\$' : ' k	0.50	250	o !	1.5 			0,20	
	SPACE SCIENCE BOARD POTABILITY LIMIT	. 20	5000 Unobjectionable Unobjectionable	3000	2000	2000 - 50 - 50	1 1	0.50		No Foem	10 15 Unobjectionabl		100	:	
	8 6/12/70 .5 gal 6/1	01 02 04 04	V V V	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	' '	!	^ ^ ^ 0.1	1	1 0 1 0 0 0 1 0 0 1 0 0 0 1 0 0 0 1 0 0 0 1 0 0 0 1 0 0 0 1 0 0 0 1 0 0 0 1 0 0 0 0 1 0 0 0 0 1 0 0 0 0 0 1 0		_+ 25 < 5 none	6.0			rporation
	6/12/70 •5 gal 5/27	> > 10 20 20	 01 01 01 01	v v v v v v v v v v	# # # # # # # # # # # # # # # # # # #		, , , , , , , , , , , , , , , , , , ,	. "	5.1.		+ 25 < 5 - none	8.7.			erojet-General Co
SES	6/12/70 5/28 5/22	× × 20	388231	2 - 2 - 2 - 2 - 2 - 2 - 2 - 2 - 2 - 2 -	285285 28785		v viv vi	v	0.4 0	. 0.6 6.3.8	+ 25 - 1 5 - 1 5 - 1 5 - 1	6.5	100		Analyses performed by Aerojet-General Corporation
WRS DETAILED PRODUCT WATER CHEMICAL AND FHYSICAL AVALYSES	5 • 5 gal 5/18	2 8 2 8	 		 	:	^ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	0.01	^ ^ ^ ^ ^ ^ ^ ^ ^ ^	. "	<pre> < 25</pre>	! ! ! ∄: ! : :			Analys
WATER CHEMICAL	4 5 gal 5/14	^ ^ 8	V: V	.v.v.v			<pre></pre>	- 1	# F	9.35	< 25 < 5 < 1 almond	51			
DETAILED PRODUCT	3 .5 gal 5/11	, v 20 20	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \		 	:	> > > > 1	1	- × ×		< 25 5 fecal	202			from ble copy.
TABLE V - WRS	2 .5 gal 5/6	> > 01 02 02 02 02 02 02 02 02 02 02 02 02 02	* P P P P P P P P P P P P P P P P P P P	2,6,8,5,2,5,4,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5	: 288888 2	:	> > > > 1 1 0 0 1 1 1 1 1		0.12	73.2	< 25 - 10		! 		Reproduced from best available co
	1 .5 gal 5/2	. v ·	: v'	1 1	/ v v v 'v v _i	:	<pre></pre>	0°00 7°00 7°00 7°00 7°00 7°00 7°00 7°00	2 2 2 2 3 0 14 0 14	14.95	< 25 < 5 aldehydic		 - -		· • • • • • • • • • • • • • • • • • • •
	SAMPLE NO. DATE (Analyses) AMOUNT DATE (Collection)	COMEOUND OR PROPERTY 2.INC CADMIUM	BORON TROS TROS TROS TROS NOT, YBDENUM NOT, YBDENUM NANGANESE	ALUMINGM PEB BENTLLIM PEB SILVER PEB SILVER PEB SILVER PEB		MERCURY FLUORIDE SELENIUM CYANIDE	1 CALCIUM (Ca) mg/1 2 MAGNESIUM (Mg) mg/1 mg/1 4 POTASSIUM (K) mg/1 mg/1 mg/1 mg/1	ARSENIC (As) AMMONIA (NH ₃ /N)	SOLFATE (SOL) "MITTERTE (NO.) "MITTERTE (NO.) TOTAL PHOSPHATE (PO.)	ABS pH COMDUCTIVITY	TUREIDITY COLOR DOOR-	TOTAL HARDNESS (G TOTAL ALKALINITY TOTAL SOLIDS CHEM O. DEMAND	UREA TOTAL PLATE COUNT TOTAL CARBON		* ½mhos ** Jacken Inits *** Threshold Odor No.
				ппн	はせおおば	~ 0 0 0 0		٦'	4 00 m 4	-17.0		~∞ o o	មាដដ	à .	

Analyses of the 6th, 7th and 8th samples sent to Aerojet-General indicate a positive turbidity value. The probable cause of appearance of turbidity in these samples is the time lag in performing the analyses after the sample was collected. The daily in-house water analyses which were usually performed on the same day the sample was collected consistently indicated a good appearance during the entire test.

The bacteriological test results shown in Table VI, indicate that the Coliform Test was negative for all samples. As previously described in the In-House Water-Quality Analyses Procedures the viable bacterial density was also determined by a Most Probable Number method. Based on the upper 95% confidence limit as given in the Standard Methods text, all samples conform to the "essential sterility" criterion of a maximum of 10 viable organisms per milliliter.

TABLE VI - WRS IN-HOUSE PRODUCT WATER MICROBIOLOGICAL ANALYSES

POTABILITY LIMIT	VIABLE BACTERIA	COLIFORM TEST	
Space Science Board	10 organisms per ml		
U.S. Public Health Service		2.2 MPN per 100 ml	

		VIABLE BAC	TERIAL DENSIT	COLIFORM		
Date	MPN PER 100 ml	95% CONFIDE Lower	NCE LIMITS Upper	MAX° VIABLE BACTERIAL DEN. (Organisms per	(MPN per	REMARKS
4-29	< 2	0	5.9	0.059	< 2	,
4-30	< 2	0	5.9	0.059	< 2	į
5-1	< 2	0	5.9	0.059	< 2	1
5-2	< 2	0	5•9	0.059	< 2	
5-3	< 2	. 0	5-9	0.059	< 2	
5-4	< 2	0	5-9	0.059	< 2	
5 - 5	< 2	ó	5-9	0.059	< 2	
5 -6	23	7	7 0	0.7	< 2 ·	—Water line opened to clean
5-7	2	< 0.5	7	0.07	< 2	check valve ~
5 - 8	< 2	0	5-9	0.059	< 2	•
5-9	< 2	0	5.9	0.059	< 2	- · · ·
5-10	< 2	0	5• 9	0.059	< 2	
5-11	4	< 0.5	11	0.11	< 2	Water line opened to replace
5-12	< 2	0	5• 9	0.059	< 2	pump diaphragm
5-13	2	< 0.5	7	0.07	< 2	
5-14	< 2	0	5.9	0.059	< 2	
5 - 15	< 2	0	5-9	0.059	< 2	
5-16	< 2	0	5.9	0,059	< 2	
5-17	< 2		5-9	0.059	< 2	=
5 - 18	2	< 0.5	7	0.07	. < 2	
5 -1 9	< 2	0	5.9	0.059	< 2	
5-20	< 2	0	5.9	0.059	< 2	
5-21	< 2		5.9	0.059	< 2	· · · · · · · · · · · · · · · · · · ·
5-22	2	< 0.5	7	0.07	< 2	·
5-23	2	< 0.5	7	0.07	< 2	
5-24	< 2	0	5.9	0,059	< 2	
5 - 25	33	11	93	0.93	< 2	Bacteria analysis sample
5-26	17	. 5	46	0.46	< 2	bottle contaminated
5 - 27	2	< 0.5	7	0.07	< 2	
5-28	11	2	25 .	0.25	< 2	
5 - 29	8	1	19	0.19	< 2	· ·· · · · -
5-30	27	11	93	0.93	< 2	
5-31	350	120	1000	10.0	< 2	Refrigerated for 5 days be-
6-1	240	68	7 50	7•5	< 2	fore performing bacterial analysis
5-1*	2	< 0.5 .	7	0.07	< 2 ·	
5-12*	< 2		5.9	0.059	< 2	
5-14*	< 2	0	5-9	0,059	< 2	· · · · -
5-19*	< 2	. 0	5.9	0.059	< 2	
5-27*	79	25	190	1.9	< 2	

^{*} Sample collected from water storage tank.
All other samples collected upstream of tank.

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

Based on the fabrication, testing and operation of the water reclamation system, the following conclusions have been reached:

- The combined process of vacuum distillation, vapor filtration, vapor phase catalytic oxidation and subsequent condensation of the product vapor are a sound approach to converting pretreated urine and respiration and perspiration condensate into potable water.
- 2. The product water conformed to the essential requirements of the U.S. Public Health Service (1962) and the National Academy of Sciences, "Space Science Board, Ad Hoc Panel on Water Quality Standards for Long-Duration Manned Space Missions", dated September, 1967. The viable bacterial density was shown to be below 1 organism per ml for all of the thirty-day system performance except for the last two days, when the test samples were refrigerated for 5 days before bacterial analyses were performed.
- 3. The ammonia content in some samples of the final product water was higher than the desired level of 10 ppm. A review of the overall test operating conditions indicated that the high ammonia content was caused by localized hot spots in the wicking material used to constrain the evaporating liquid for weightless operation.
- 4. A rotating bowl for separating collected urine from pneumatic transport gas was shown to be feasible, with the design suitable for weightless operation. For the configuration of a rotating bowl with stationary pitot tube, addition of an antifoam agent to the urine prevents foaming within the bowl.

- 5. Storage tanks which retain liquid between the bladder and tank wall were shown to be practical for storing liquids and for utilizing gas pressure within the bladder to expell liquid from the tank; this design is also suitable for weightless operation.
- 6. A vapor permeable membrane in the evaporator still has a limited useful life as a liquid-vapor phase separator when liquid and solid residues contact the membrane. Verification tests with urine liquid and liquid and solid residue contacting the membrane indicated that approximately one week of operation is achieved before direct leakage of residue and vapors through the membrane occurs.
- 7. A rotating phase separator at the condensing coil discharge was shown to be practical for separating product condensate from vapors and non-condensibles. Performance tests in normal gravity indicated that approximately 1% of the process vapor along with non-condensibles is lost to vacuum. The phase separator transferred water at a pressure head 6 in. WG above the intake level. Water transfer out of the separator is thus accomplished by the dynamic head of the rotating liquid impinging on a stationary pitot type collection tube.
- 8. Evaporation of urine from the wicks is feasible, but sustained liquid flow into the wick and control of heat transfer to and through the wicks is critical. The tendency of the wicks to become clogged by depositing solids along the immediate heat input surface suggests that an extended and uniform heat transfer surface should be used to prevent local hot spots in the wick.

- 9. The method of utilizing two equal displacement peristaltic pumps driven by a common motor to transfer equal amounts of urine into, and potable water out of the system, to regulate the liquid level in the evaporator was determined to be inadequate for accurate control. The inaccuracy resulted from cavitation at the water pump intake resulting in a sporadic and pulsating water removal rate, while the urine feed rate remained continuous and stable.
- 10. A similar approach to provide equal flows, but with two equal displacement diaphragm pumps driven by a common motor, also was inadequate for accurate level control. The inaccuracy resulted from inequalities in the opening pressure differential for the check valves in the pump heads which, in turn, caused unequal flows from each head.
- ll. A feed control device which benefits from the pressure differential between the waste holding tank and evaporator still is practical and was observed to operate consistently and with repeatable metering accuracy during tests. A mechanically timed method of controlling the feed rate was shown to be practical for this application where constant level thermal power input is provided.
- 12. A dual head diaphragm pump for transferring the product water from condensation vacuum to storage tank pressure was determined to be practical and to be the perferred method over other pumping methods. The dual head arrangement requires maximum torque at 180° offset intervals, thereby allowing a higher efficiency than with a single pump head.

- 13. An electro-mechanical sensor and control which functions similarly to a differential pressure switch was determined to be a sound approach for controlling the amount of water in the condenser receiver. Because only a low-pressure differential is available to actuate the control, its orientation is restricted for operation in normal gravity. Since it is mechanically simple and consumes no power, it is perferred over electronic type sensors.

 By comparison, a conductivity sensor in the evaporator still used for a test backup control failed within 48 hours after the start of the test.
- 14. Small brushless DC motors were observed during tests to operate without interruption or the need for corrective maintenance, as might otherwise be required for brush-type motors.
- 15. A magnetic coupling which transmits motor torque from the ambient environment to the condenser phase separator vacuum environment was shown to operate satisfactorily. The inertia of the separator wheel inhibited rapid start-up from rest; startup at reduced motor speed followed by a gradual increase to normal speeds was required to maintain proper torque transmission.
- 16. Type 316 stainless steel provides satisfactory corrosion resistance for use in the evaporator still.
- 17. Type 316 stainless steel is attacked by the ARDOX catalyst and/or its by-products for a short time after the catalyst is installed in the housing. An analysis of the attacked surfaces indicated the major cause of attack was by bromine or its derivatives.

18. Material compatibility tests to determine suitable elastomers for use in the system with the 4:1:4 mixture of H₂SO₄, CrO₃ and H₂O pretreatment solution indicated that acid resistant Viton o-rings manufactured by Parker Seal Co., and Hypalon rolling diaphragms manufactured by Bellofram Corp., are suitable. Viton diaphragms were shown to be unresistant to acid attack.

RECOMMENDATIONS

Based on the observations drawn from this program, the following recommendations are made concerning further development of a vacuum distillation-vapor filtered-catalytic oxidation water reclamation system.

- Low temperature catalysts should be developed which will also reduce ammonia content as well as organic compounds in the product vapor. Long term tests should be conducted to further define proper operating conditions and useful life of the catalyst used during performance tests.
- 2. Pretreatment chemicals are expended during system operation.

 Further development effort should be conducted to determine the optimum quantity of pretreatment so that the system will require a minimum weight of expendables.
- 3. Evaporator still phase separation techniques should be further investigated with respect to the final disposition of the accumulated residue and with respect to the separation of generated vapor from distillation liquid. A suggested approach for retaining the residue within the evaporator still is to utilize a volume of wick sufficient to hold the residue which accumulates during the desired operating life of one still. The wick volume should be proportioned to have

an approximate 2-inch vertical height to be compatible with testing in normal gravity. A suggested alternate approach for vapor-liquid separation, which also allows for periodic removal of residue from the evaporator still, is to utilize centrifugal action within the evaporator. This method is compatible with periodic or continuous removal of residue without disassembly of the still or repeated flushing to remove the residue.

- 4. If the product water is to be stored for longer than one or two days, means should be provided to re-sterilize the water before it is consumed. Tests indicate that bacterial growth after five days of refrigerated storage led to viable organism levels approaching the allowed limit for potable water.
- 5. Instead of maintaining a specified volume of liquid in the evaporator to keep all heating surfaces covered and to prevent localized hot-spots, alternate methods should be investigated for utilizing heat inputs and controlling the heat transfer surface temperature. One probable method is to use an intermediate fluid loop for heat transport between the heat source and the evaporator still.
- 6. Storage tanks for urine, flush water, pretreatment liquid, and potable water should be constructed to incorporate an empty-to-full indicator so that the volume in storage will be known at any given time.